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<p>LiZn ferrites were investigated to control the resultant microstructure or to obtain nanoscale powders at a low temperature. Two nonconventional chemical methods are introduced. The sol-gel coating procedure was used to uniformly distribute additives like SiO_2 and Bi_2O_3. These oxides improved the densification of the LiZn ferrites at low temperatures via a liquid phase sintering. Nanocrystalline LiZn ferrites were obtained at a low temperature of 400°C via the assistance of a combustible acid like polyacrylic acid.</p>			
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**DEVELOPMENT OF MICROWAVE FERRITES FOR HIGH
PERFORMANCE APPLICATIONS**

TECHNICAL REPORT

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Development of Microwave Ferrites for High Performance Applications

Two chemical methods applied to LiZn ferrites will be briefly introduced. The influences of the experimental parameters and compositions on the final microstructures and properties are being investigated. Several reprints and manuscripts resulted from the previous research on yttrium iron garnet materials are enclosed (reported in technical report submitted to ARO on 2/5/96).

I. Microstructural Control of LiZn Ferrites with Chemically Derived Additives

Raw materials such as Li_2CO_3 , ZnO and Fe_2O_3 were mixed using yttria stabilized zirconia balls (Tosoh Co.) and calcined at 800°C for 4 hrs to form a spinel phase. Two additives, Bi_2O_3 - MnO_2 and SiO_2 - MnO_2 were incorporated into $\text{Li}_{0.3}\text{Zn}_{0.4}\text{Fe}_{2.3}\text{O}_4$ via the sol-gel coating method. The calcined powders were milled using the same media. The experimental procedures for sol-gel coating are as follows. In case of Bi_2O_3 - MnO_2 coating, Bi nitrate and Mn acetate were used as raw materials. Bi nitrate pentahydrate, $\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$ and Mn acetate tetrahydrate, $\text{Mn}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$ corresponding to 1 wt% Bi_2O_3 and 1 wt% MnO_2 were dissolved completely into deionized water. A 50 wt% aqueous solution of polyacrylic acid (PAA) was added to the solution resulting in a gelatinous white precipitate. And then the calcined LiZn powder was mixed with the gel. For the SiO_2 - MnO_2 coating, tetraethylorthosilicates (TEOS), $\text{Si}(\text{OC}_2\text{H}_5)_4$ and Mn acetate tetrahydrate, $\text{Mn}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$ corresponding to 1 wt% SiO_2 and 1 wt% MnO_2 were dissolved into ethanol. A few drops of HCl was added as a catalyst for sol-gel reaction. The calcined powder of LiZn ferrite was added into the clear solution and kept stirring for a few hours for complete reaction. All coated powders were dried at 70°C and then pulverized using a mortar. Pellets were made by uniaxial pressing of the powder at around 12,000 psi and sintered at 1050°C for 2 hrs with a heating rate of $300^\circ\text{C}/\text{hr}$ after organic burnout at 500°C .

The preliminary observation of microstructures showed that Bi_2O_3 and SiO_2 enhanced grain growth and densification. Large grains were observed in these cases. Distinct grain boundary regions were seen after chemical etching in case of Bi_2O_3 , indicating a weak grain boundary layer. While, in case of SiO_2 , it is likely that the addition SiO_2 induces a glassy phase in the grain boundaries (not easily etchable). Based on the grain size variations with temperature and time, grain growth kinetics will be studied for the different compositions. Magnetic and dielectric properties in the microwave frequency

range will be also investigated. Additionally, different contents of the additives will be adjusted in order to optimize the magnetic and dielectric properties, .

II. Preparation of Nanocrystalline LiZn ferrites Using Polyacrylic Acid

A new chemical method utilizing a combustible polyacrylic acid (PAA) is introduced to prepare nanoscale ferrite powders. This method is relatively simple compared to other chemical methods. Nitrates of Li, Zn and Fe were dissolved in deionized water to form a 0.5 M solution. The nitrate solution was inserted into a 50 wt% polyacrylic acid aqueous solution. The atomic ratio of the carboxyl ion to cation was 0.5. After a few minutes, the clear nitrate solution changed into an opaque viscous gel having brown color. The gel was dried at 70°C and calcined from 400°C to 700°C for 30 min.

As a result of TEM observation, the average particle size of the LiZn ferrites fired at 400°C was around 10 nm. The XRD pattern indicated the crystalline LiZn ferrite phase. As the synthesis temperature increases, the particle size tended to increase but the nanoscale was kept until 600°C. From the initial results, it can be concluded that the PAA method is promising for the preparation of nanoscale LiZn ferrite powders at low temperatures. Densification behavior of the nanosized ferrites will be investigated.

List of Publications

- Y. S. Cho, V. L. Burdick and V. R. W. Amarakoon, "Hydrothermal Preparation and Morphology Characteristics of $Y_3Fe_5O_{12}$," *J. Am. Ceram. Soc.* (submitted, Paper No. 191632C)).
- Y. S. Cho, D. T. Hoelzer, L. Brissette and V. R. W. Amarakoon, "Grain Boundary and Electrical Characteristics of Chemically Modified Gd-YIG," *J. Mater. Res.* (submitted, Paper No. 96-271).
- Y. S. Cho and V. R. W. Amarakoon, "Nanoscale Coating of Silicon and Manganese on Ferrimagnetic Yttrium Iron Garnets," *J. Am. Ceram. Soc.*, **79**(10) 2755-58 (1996).
- Y. S. Cho, L. Brissette and V. R. W. Amarakoone, "Effect of Sol-Gel Coating on the Microstructure and Electrical Properties of Microwave Garnets," *Mater. Res. Soc. Symp. Proc., Vol. 411, Electrically Based Microstructural Characterization*, pp287-292 (1996).

List of Presentation

- Preparation of Nanocrystalline Ferrites via the Assistance of Polyacrylic Acid,, *The 99th Annual Meeting of Am Ceram. Soc.*, Cincinnati, OH, May 1997.
- "Grain Growth and Microstructural Characteristics of LiZn Ferrites with Chemically Derived Additives," *The Fall Meeting of Mater. Res. Soc.*, Boston, MA, Dec. 1996.
- Hydrothermal Synthesis of Ferrimagnetic Yttrium Iron Garnets, *The 98h Annual Meeting of Am Ceram. Soc.*, Indianapolis, IN, Apr. 1996.
- Effect of Sol-Gel Coating on the Microstructure and Properties of Microwave Garnets, *The Fall Meeting of Mater. Res. Soc.*, Boston, MA, Nov. 1995.
- Microstructure and Phase Evolution in Ferrimagnetic Garnets Prepared with Additives via Sol-Gel Coating, *The 97th Annual Meeting of Am Ceram. Soc.*, Cincinnati, OH, May 1995.